NSSI Neutron Newsletter

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Neutron Scattering Society of India

Contents

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The President's Column

I am glad that the first issue of NSSI Newsletter is being published. It is aimed to showcase the activities of NSSI and include some important research articles and general news on neutron scattering. I would like to congratulate the editorial team for their efforts for taking the responsibility

For the last six decades the thermal neutron have served as an indispensable probe of the structure, magnetism and dynamic in condensed matter, providing very important and useful information that is complementary to that observed using X-rays and synchrotrone radiation. In India, neutron scattering program started in the late 1950s and has been centered around the Apsara, CIRUS and Dhruva reactors at Bhabha Atomic Research Centre (BARC). Presently, a diverse program in condensed matter research is pursued at the Dhruva reactor, which has been aided by both national and international collaborations. For over two decades, scientists from many national institutes and universities in India have been participating in collaborative neutron scattering experiments at BARC through a scheme under which the proposals are submitted biannually to University Grants Commission-Department of Atomic Energy-Consortium for Scientific Research (UGC-DAE-CSR). A large number of collaborative projects have also been funded by BRNS and other agencies.

Neutron Scattering Society of India (NSSI), originally named as Indian Neutron Scattering Society (INSS), was formed in 2008 during a meeting of neutron users at Mumbai with an objective to promote the research and development activities of neutron-scattering science and applications. Another aim to form NSSI was to represent the neutron users in the Asia-Oceania Neutron Scattering Association (AONSA), which is an affiliation of neutron scattering societies in the Asia-Oceania region. NSSI at present has over 120 members.

NSSI as its first major endeavor was associated with the School and Conference on Neutron Scattering and Mesoscopic Systems held in Mumbai and Goa in 2009. Subsequently it co-organised the 3rd AONSA neutron school in BARC in 2010, and the 2nd International Symposium on Neutron Scattering in BARC during January 14-17, 2013. The next School cum Conference on Neutron Scattering is proposed to be held in Mumbai/Pune during February 2014.

We hope to publish the Newsletter twice yearly and welcome the news and contributions from the members and also others. Feedback from the readers is welcome and that will improve the quality of the Newsletter.

S.L. Chaplot

Neutron Scattering Society of India

(Registered No. Maharashtra State, Mumbai, 2011/GBBSD/1696)

(For Promotion of Neutron Scattering Research in India)

C/o Solid State Physics Division
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Neutron scattering society of India (NSSI) formerly known as Indian neutron scattering society has grown up considerably. It started with merely 20 members in 2008 and at present it has 120 members. Main objective of the society was to promote neutron scattering research in India and represent our country in the Asia-Oceania Neutron Scattering Association (AONSA) (http://www.aonsa.org) which is an affiliation of neutron scattering societies and committees that directly represent users in the Asia-Oceania region. The main purposes of the AONSA are to provide a platform for discussion and a focus for action in neutron scattering and related topics in the Asia-Oceania region.

Several neutron-related meetings were held in recent past in which members of NSSI were involved. This included a theme meeting at BARC in March 2012, and review meetings of neutron projects supported by UGC-DAE CSR in March and October. UGC-DAE CSR organized an awareness workshop at Gitam University, Vizag, during January 26-27, 2012. About 50 faculty and students attended the workshop. Apart from other topics, various aspects of neutron scattering along with neutron scattering facilities available at BARC were discussed. An India-ORNL workshop on Exploring Materials using Spallation Neutron Source was organized on September 6 and 7, 2012 at JNCASR, Bengaluru supported by Indo-US S&T Forum to promote collaboration at ORNL. About 40 scientists from India and about 10 from ORNL attended the meeting.

A managing committee of NSSI has members from several universities (Varanasi, Chandigarh, Goa and Delhi) and national institutions (BARC, TIFR, IITM, and UGC-DAE CSR) representing various users and the spectrum of neutron scattering activities. At present NSSI consists of members from universities and institutions spread all over the country. Two members of NSSI are in the executive committee of the AONSA, and one member is an observer. NSSI as its first major endeavor was associated with the School and Conference on Neutron Scattering and Mesoscopic Systems held in Mumbai and Goa during October 5-14, 2009. The 3rd AONSA neutron school was organized in BARC during October 4-9, 2010. A large number of participants from Asia-Oceania region (Australia, New Zealand, Japan, Korea, Taiwan, Indonesia and Malaysia) attended the school. Lecturers in the school were

from these countries apart from Indian. The last mega event organized was the 2nd International Symposiun on Neutron Scattering in BARC during January 14-17, 2013. This was preceded by the XV Workshop on Neutron as a Probe of Condensed Matter during January 8-12, 2013. The response for the workshop was overwhelming, about 130 applications were received and due to logistic reason only 60 participants were selected. The next School cum Conference on Neutron Scattering is proposed to be held in Mumbai/Pune during February 2014.

A National Facility for Neutron Beam Research (NFNBR) has been operating for more than two decades at BARC for basic and applied research in condensed matter science. The present-day facilities include, single-crystal and powder diffractometers, polarization analysis spectrometer, hi-Q diffractometer, triple-axis & filter-detector spectrometers, quasi-elastic scattering spectrometer (all installed in the reactor hall), and two small-angle scattering instruments, and a reflectometer (in the guide-tube laboratory). An instrument for residual stress analysis is under development. The National Facility is regularly utilized in collaboration with about 200 users from various universities and other academic institutions. Support for the collaborations is available from University Grants Commission-Department of Atomic Energy Consortium for Scientific Research (UGC-DAE CSR), Board of Research in Nuclear Sciences (BRNS) and other agencies in India. BARC and UGC-DAE-CSR have jointly organized many Workshops/Schools on Neutrons as Probes of Condensed Matter. These involve training in basic principles and various applications in physics, chemistry, biology and materials science, including hands-on experiments and data-analysis at Dhruva.

Participants along with the organizers gather for a group photo at foyer of the Dhruva reactor during 3rd AONSA neutron school held at BARC.

Indian participants gather for a group photograph after the regional meeting during 1st AOCNS in Tsukuba, Japan.

Dr. S.L. Chaplot, Chairman, 2nd International Symposium on Neutron Scattering (ISNS 2013) addressing during the inaugural function held in Mumbai on January 14, 2013. Shri Sekhar Basu, Director BARC, Dr S. Kailas, Director, Physics Group, BARC and Dr. S.M. Yusuf, Secretary, Local Organization, ISNS 2013 are on the dais.

Activities of AONSA are getting stronger with time. One of the most important activities of AONSA is the Neutron School, which is held once in every year since its formation in 2008. It has organized Neutron Schools, the first at Korea (2008) and the second at Australia (2009), third in India (2010), next was scheduled to be held in Japan but due to severe earthquake and Tsunami it was shifted to Australia (2011), and the last was held in China (2012). Participants were, in general, 50% from the host countries and other 50% distributed among other Asia-Oceania counties. The first Asia Oceania Conference on Neutron Scattering (AOCNS) was held in Tsukuba, Japan, in November 2011, was an extraordinary success that demonstrated the breadth and depth of AONSA's reach within the Asia-Oceania neutron community. A large number of participants from India attended the conference. During the conference regional meeting of all the member societies was held. Participants from India also organized a meeting, exchanged views and discussed various suggestions regarding the activities of NSSI and improvement on usage of neutrons.

We are happy to inform the website of NSSI is now available. In this regard we thank Prof T. Pradeep for hosting the website at the IIT Madras site and also thank Dr. S. Mitra for helping out to make the website design. The website contains the rules and regulations, by-laws, list of present members and also the membership form. The website address is: http://www.nssi.iitm.ac.in. Members are requested to visit this site and their suggestions to improve the site are welcome.

We are enthusiastic about the growth of NSSI and neutron science in India.

Managing Committee of Indian Neutron Scattering Society

Prof. S. L. Chaplot, BARC, Mumbai **(President)**
Prof. R. Mukhopadhyay, BARC, Mumbai (**Secretary)**
Dr. V. Siruguri, UGC-DAE CSR, Mumbai **(Treasurer)**
Prof. H. B. Bohidar, JNU, New Delhi, Member
Prof. J.A.E. Desa, University of Goa, Goa, Member
Prof. A.K. Grover, TIFR, Mumbai, Member
Prof. V.K. Jindal, Punjab University, Member
Prof. D. Pandey, BHU, Varanasi, Member
Prof. T. Pradeep, IIT Madras, Chennai, Member

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National Facility for Neutron Beam Research

Solid State Physics Division, Bhabha Atomic Research Centre, Mumbai 400085, India

The short-range strong interaction of neutron with matter and its inherent magnetic moment make neutron scattering a unique probe in condensed matter research. The other important advantage of neutrons over other forms of radiation in the study of structure and dynamics on a microscopic level are they are uncharged, which allows them to penetrate the bulk of materials. They interact via strong nuclear force with the nuclei of the material and the scattering cross-section varies randomly between various elements and even between isotopes. This allows one to observe light atoms such as hydrogen in the presence of heavier ones and distinguish neighbouring elements in the periodic table easily. One can exploit isotopic substitution and contrast variation methods.

Matching wavelength and energy of thermal neutron to the lattice spacing and excitations in condensed matter makes it an indispensible tool to both study structure and dynamics. Over and above neutron has a magnetic moment that suits to study magnetic structures and the fluctuations and excitations of spin systems.

Presently, a National Facility for Neutron Beam Research (NFNBR) is operated by Solid State Physics Division (SSPD), Bhabha Atomic Research Centre (BARC), Mumbai, for the use of neutrons in condensed matter research. NFNBR has been built around research reactor Dhruva. Dhruva is a natural uranium based thermal reactor. The peak thermal neutron flux of Dhruva reactor is about 1.8×10^{14} neutrons/cm^2/s, when it is operated at its maximum power of 100 MW.

The neutron scattering facilities are extensively used by SSPD scientist as well as by a large number of other users. Since around 1990, after the Inter-University Consortium for the Department of Atomic Energy Facilities (IUC-DAEF) came into existence, an increasing number of scientists from the national institutes and universities in India have been formally carrying out collaborative neutron scattering experiments at BARC. IUC-DAEF renamed recently as University Grants Commission-Department of Atomic Energy Consortium for Scientific Research (UGC-DAE CSR) mediates the use of NFNBR programme for the university researchers. Periodically, almost every year, UGC-DAE CSR and Solid State Physics Division of BARC have been conducting joint workshops and schools on different aspects of neutron diffraction and scattering techniques. Some 15 workshops and schools have been held since 1989 and more than 500 researchers have received training in applying neutron scattering techniques in their own areas of research. The university system has served greatly by augmenting the availability of physically and technologically interesting materials for the experiments. To enhance the university participation the UGC-DAE CSR has more

recently installed a neutron beam line at Dhruva. Several university groups have actively contributed to the instrumentation effort by developing and building certain identified components of the beamline. At present there are about 35 active researcher projects from universities or national institutions using NFNBR under the UGC-DAE CSR programme.

Plan layout of the beam lines at the Dhruva reactor and the neutron scattering instruments in the reactor hall and guide laboratory are shown in fig. 1 and the photograph of the panaromic view of the neutron scattering facility at Dhruva is shown in fig. 2.

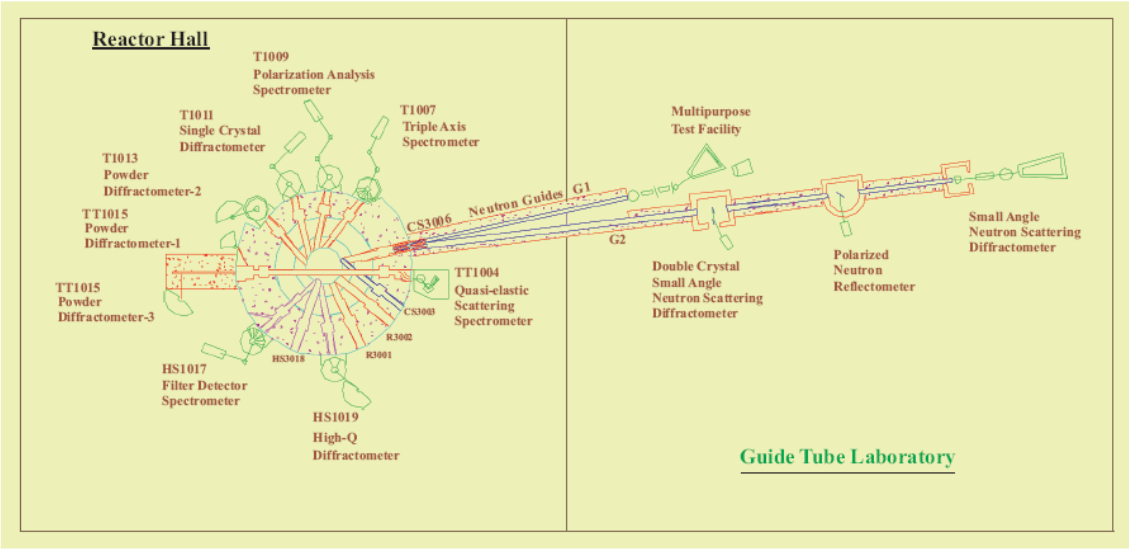

Fig. 1 Plan layout of the neutron scattering facility at Dhruva, BARC, Trombay

Fig. 2. Panaromic view of the neutron instruments at Dhruva. The instruments seen are, from left, triple axis spectrometer, polarisation analysis spectrometer, single crystal diffractometer, multi-position sensitive detector (PSD) based powder diffractometer, on the four tangential beamtubes, a single PSD based powder diffractometer at the end of a through-tube and filter detector spectrometer at the extreme right. The yellow coloured block is basically the shield of the instrument developed by the university researcher under UGC-DAE CSR. The hi-Q diffractometer and quasielastic spectrometer are on the opposite side and not seen in the photo. The guide tube laboratory is on the left of the reactor hall, the shielding of the guide is seen.

At present a four-circle single-crystal diffractometer, three powder diffractometers, a high-Q diffractometer, a polarization analysis spectrometer, a triple-axis spectrometer, a filter detector spectrometer, and a quasi-elastic scattering spectrometer are located inside the reactor hall on various beam ports (fig. 1). The powder diffractometer-3 is installed in the reactor hall by UGC-DAE CSR.

Two neutron guide tubes (details given in Table 1), G1 and G2 (length: 21m and 35m, radius of curvature: 1916m and 3452m, characteristic wavelength: 3.0Å and 2.2Å respectively) transport neutron beams in to Guide-Tube Laboratory from the reactor hall (fig. 3). Average flux at the breaks, provided on the guides to accommodate various instruments, is about 10^7 neutrons/cm^2/s. Two small-angle neutron scattering instruments and a polarized neutron reflectometer are operational at G2 one after another (fig. 3). The beam port at the guide G1 is used for the testing of detectors.

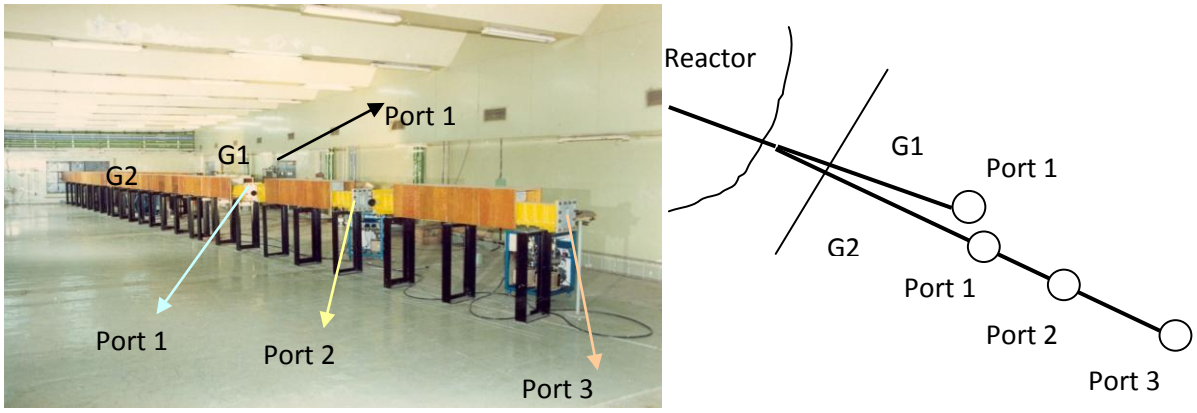

Fig. 3. The neutron guides as were laid in the guide tube laboratory (before the shieldings were put around), only G1, the longer guide (35 m from the reactor face) is seen, G2, the shorter one (21 meter) is on the right side of G1.

Table 1. Characteristics of Curved Neutron Guide Tubes Installed at Dhruva

Physical Property	**Guide 1**	**Guide 2**
Critical Wavelength	3.0 Å	2.2 Å
Radius of Curvature	1916 metres	3452 metres
Length of Direct Vision	20 metres	27 metres
Total Length	21 metres	35 metres
No. of Experimental Posts	1	3
Neutron flux (10^7 neutrons/cm^2/sec)	1.4	2.9, 2.0, 1.7
Beam Size	25 mm × 100 mm	

In addition, for the engineering applications of neutron an instrument is under developement to study the residual stress in materials. Neutron is an ideal probe to estimate the stress developed in bulk objects subjected to constant or repeated stress. The absorption cross section for neutrons in materials of technological interest such as Fe and Al is very low. Therefore, it penetrates deep into the bulk of object to provide information about local deformations. This instrument therefore would provide a unique opportunity to study stresses developed deep inside a material. Objects under study could be welded joints, rolled sheets or plates, defects in metal composites and other similar projects.

All the neutron instruments, guide tubes, detectors, etc., have been designed and developed in-house by neutron beam researchers in SSPD. Fabrication, installation and testing of them, as well as the development of instrument control and data acquizition systems, have been carried out in collaboration with various Divisions in BARC. These facilities are continuously upgraded and maintained to facilitate research on materials of scientific interest and technological importance. NFNBR develops and fabricates neutron detectors, for both in-house use and supply to other users in the country, using BF_3 and He^3 gases. Linear position-sensitive detectors, using He^3 gas have been developed and extensively used at BARC during the past decade and has given a tremendous impetus towards data acquisition in terms of increased through-put.

As stated before due to the unique properties of neutron, it can be used to study structure in different length scales, both chemical and magnetic, dynamics in a wide energy range. A variety of instruments are built to explore different aspect of condensed matter physics, chemistry, biology and material science.

To summarize, the success of Indian neutron scattering program has depended much on building indigenously the massive neutron spectrometers, detectors and other associated systems, which would have been prohibitively expensive to procure from abroad. In particular, standard and one-dimensional position sensitive detectors and associated electronics are now routinely produced in-house and finally from ECIL. We have used neutron scattering extensively to explore different aspect of condensed matter physics, chemistry, biology and material science by studying structures in different length scales and dynamics in a wide energy range. National collaborations through the UGC-DAE CSR have helped tremendously to enhance neutron scattering based research in the country.International collaborations are helping to keep apace with the fast developments worldwide.

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Report on
School on Neutrons as Probes of Condensed Matter (XV-NPCM-2013)

January 8 – 12, 2013, BARC, Mumbai

UGC-DAE Consortium for Scientific Research (CSR), Mumbai centre in association with Solid State Physics Division (SSPD) BARC organized a five day school on Neutrons as Probes of Condensed Matter (NPCM) from January 8 – 12, 2013 at BARC, Mumbai, which was fifteenth in this series of schools / workshops organized by CSR in association with BARC in highlighting the use of neutrons in condensed matter physics. This edition of the school was organized as a satellite workshop to the second International Symposium on Neutron Scattering (ISNS-2013) organized by DAE and BRNS during Jan 14 – 17, 2013. The XV-School on Neutrons as Probes of Condensed Matter (NPCM) was coordinated by Dr. Sudhindra Rayaprol (UGC-DAE CSR, Mumbai Centre) and Dr. Surendra Singh (SSPD, BARC).

The school had about 60 participants of which 20 were faculty members and 40 were research students from Indian universities and research institutions. The participants were from almost all parts of India, farthest being Tezpur in the East, Chennai in South, Hamirpur in the north and Rajkot in the west.

A group photo of participants along with the organizers during the neuron school

The school comprised three days of lectures/tutorials and two days of experiments at Dhruva reactor, BARC. Eleven lectures covering various aspects of neutron scattering were delivered by invited speakers on the first two days of the school. All participants were taken inside BARC during the next two days for experimental sessions at Dhruva reactor. Participants were divided into 10 groups, and each group participated in two experiments over these two days. All the neutron scattering instruments available under the National Facility for Neutron Beam Research (NFNBR) were utilized for the experimental sessions. On the last day of the school, tutorials on neutron diffraction data analysis and small angle neutron scattering (SANS) data analysis were conducted.

The school started with the welcome remarks by Dr. Vasudeva Siruguri (Centre Director, UGC-DAE CSR Mumbai Centre), who welcomed the participants and briefed them about the mandate and activities of UGC-DAE CSR, especially Mumbai centre. Dr. S.L. Chaplot (Head, SSPD, BARC) also welcomed the participants and elucidated the role of NFNBR in promoting research based on neutron scattering in India. The sessions of the school consisted of the following lectures: Basics of Neutron Scattering (Anil Jain, SSPD, BARC), General Overview of NFNBR (R. Mukhopadhyay, SSPD, BARC), Neutron diffraction - Chemical Structures (Amitabh Das, SSPD, BARC), Small Angle Neutron Scattering - I: Basics, Theory and Applications (V. K. Aswal, SSPD, BARC), Small Angle Neutron Scattering - II (Debasis Sen, SSPD, BARC), Polarized Neutron Reflectometry (Surendra Singh, SSPD, BARC), Neutron Diffraction - Magnetic Structures (P.D. Babu, UGC-DAE CSR, Mumbai Centre), Liquids and Amorphous Systems (P.S.R. Krishna, SSPD, BARC), Single Crystal Diffraction (R. Chitra, SSPD, BARC), Inelastic Neutron Scattering (Mala N. Rao, SSPD, BARC) and Quasi-Elastic Neutron Scattering (V.K. Sharma, SSPD, BARC). The following tutorials sessions were conducted: Rietveld Refinement Method using Fullprof Suite (S. Rayaprol, UGC-DAE CSR, Mumbai Centre), Rietveld Refinement Method for Magnetic Scattering (Amit Kumar, SSPD, BARC), and SANS Data Analysis (Sugam Kumar, SSPD, BARC). After the tutorial sessions, a few participants presented their research work and also mentioned how they plan to use neutron scattering in their research activities.

Participants attending a lecture during the neutron school

Certificates of participation were given to all participants. Participants were invited to give their feedback about the school. The school was well received by all participants, many of them being first-timers to neutron scattering, and they expressed their happiness in participating in this school.

Report on
2nd International Symposium on Neutron Scattering (ISNS 2013)

January 14 – 17, 2013, BARC, Mumbai

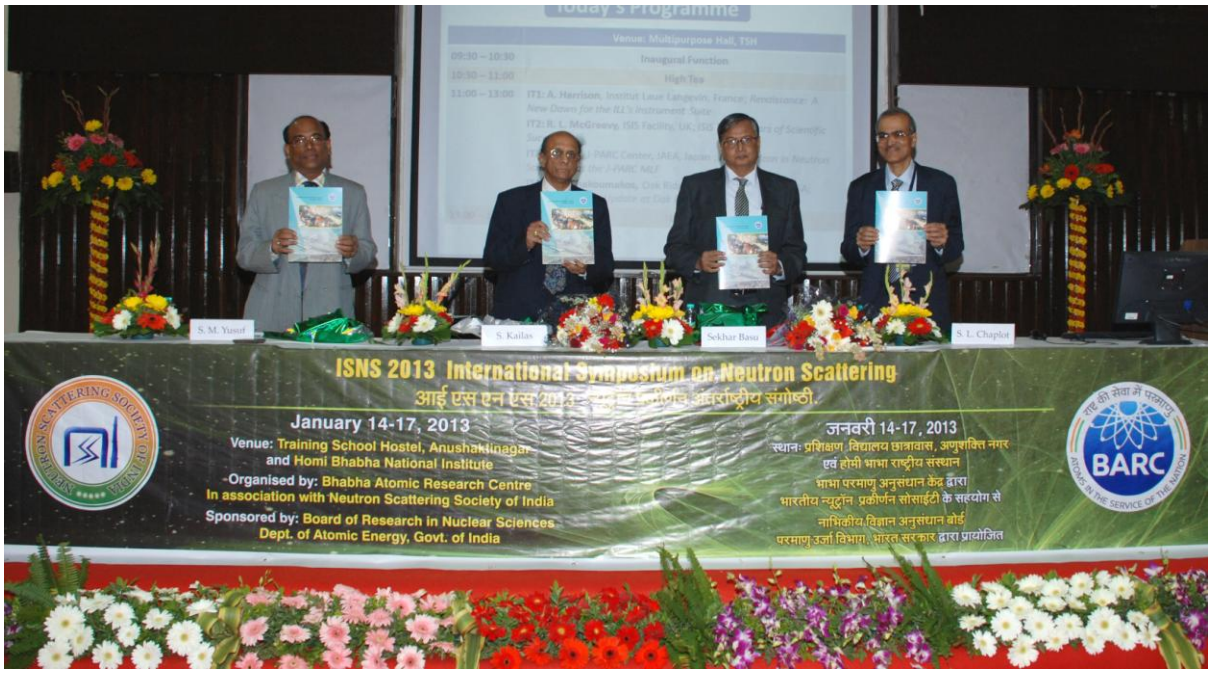
The 2nd International Symposium on Neutron Scattering (ISNS 2013), sponsored by Board of Research in Nuclear Sciences, India, and was organized by BARC in association with Neutron Scattering Society of India, was held at Training School Complex at Anushaktinagar Mumbai, during Jan 14-17, 2013. The first ISNS was held at Mumbai in January 2008. The symposium was inaugurated by Shri Sekhar Basu, Director, BARC and Dr. S. Kailas, Director, Physics Group BARC presided over the inaugural function.

The symposium covered all aspects including neutron scattering facilities, instruments, science and applications. Particular emphasis was on application of neutrons in studies of energy storage, batteries, functional materials and soft matter, besides simulation of experiments and detectors.

For ISNS there were more than 200 participants including several world leaders from laboratories in Australia, Belgium, France, Germany, Japan, Korea, Poland, Russia, Spain, Switzerland, UK, USA, and others. There were 50 invited talks, 18 oral presentations and 87 poster presentations. Within the invited talks 13 facility reports were in the plenary session. The facility reports covered almost all present day mega-facilities like, ILL, Grenoble, France, ISIS facility (UK), J-PARC (Japan), ORNL (US), FRM II (Germany), SINQ (PSI, Switzerland), LLB (Saclay, France), JINR (Russia), ANSTO (Australia), JCNS (Juelich, Germany), KAERI (Korea) and BARC (Mumbai, India). The upcoming facility European Spallation Source at Lund was also presented. The other 37 talks were in parallel sessions, which covered the wide range of science that is being pursued all over the world. In all 87 contributed papers were put up as posters, distributed on two days.

Researchers from various universities and other academic institutions utilize the National Facility for Neutron Beam Research at BARC regularly. This international symposium enabled very useful scientific discussions among the national and international researchers. A visit was organized to the neutron scattering facility at the Dhruva reactor. Overall it was a very successful symposium and participants expressed their happiness for both the academic and social aspects.

A pre-symposium school on “neutron as probes of condensed matter” was also organized for the researchers in the national level during Jan 8-12, 2013, which included hands-on experiment along with the theory classes. The response for the school was overwhelming, but no more than 60 participants could be considered for logistic reasons.



Release of the brochure on NFNBR during the inaugural function of the ISNS 2013. From left, Dr. S.M Yusuf, Local Convener, ISNS 2013, Dr. S. Kailas, Director, Physics Group, BARC, Shri Shekhar Basu, Director, BARC and Dr. S.L. Chaplot, Head, SSPD, BARC and Chairman Organising Committee ISNS 2013, are on the dais.

Shri Shekhar Basu, Director, BARC, delivering inaugural address during the symposium.

Participants attending sessions during the symposium

Investigation of Interface Magnetism using Polarized Neutron Reflectivity

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Solid State Physics Division, Bhabha Atomic Research Centre, Mumbai 40085

A rapidly increasing number of research and applications in science and engineering rely on thin films, multilayers down to the sub-nanometer scale. Modern synthesis methods have yielded high-quality heterostructures. Interfaces between two materials can give rise to novel physical phenomena and functionalities not exhibited by either of the constituent materials alone. Thus interfaces are expected to be important in determining the macroscopic properties of such systems. In order to understand the physical, magnetic properties of interfaces in nanostructures, a thorough and detailed structural and magnetic characterization is required. X-ray reflectivity (XRR) and polarized neutron reflectivity (PNR) are two nondestructive techniques that provide quantitative measures of the chemical and magnetic depth profiles of films with less than nanometer resolution [1-5] averaged over the lateral dimensions of the entire sample (typically 100 mm^2).

Reflectivity (XRR or PNR) involves measurement of the x-ray/neutron radiation reflected from a sample (Fig. 1) as a function of wave vector transfer Q (i.e., the difference between the outgoing and incoming wave vectors). The most intensely reflected beam (thick black arrow Fig. 1a) corresponds to the specular reflectivity where the angle of reflection from the surface θ_f and the angle of incidence θ_i are equal. In addition, off-specular scattered (diffuse) radiation i.e., data in a range of θ_f for a single θ_i (thin blue arrows, Fig. 1a) [6] gives roughness in terms of height-height correlation along the lateral dimensions of the sample on micron length scales.

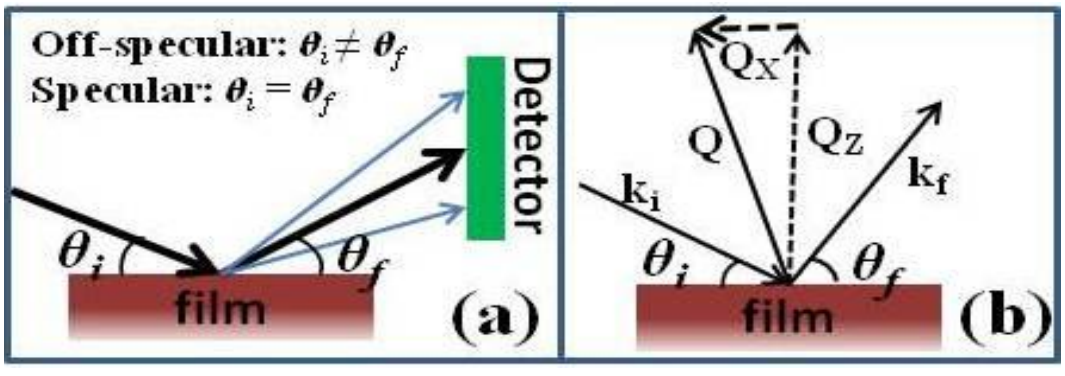

Fig. 1 (a): Neutron/x-ray scattering geometry using position sensitive detector. (b) Scattering geometry in reciprocal space.

Specular reflectivity (angle of incidence = angle of reflection) involves measurement of the radiation (x-ray /neutron) reflected from a sample as a function of wave vector transfer Q_z [= 4πsinθ/λ], perpendicular to the sample surface where λ is x-ray or neutron wavelength and θ angle of incidence. The specular reflectivity is quantitatively related to the Fourier transform of the scattering length density (SLD) depth profile $\rho(z)$ [3, 4], averaged over the sample area. For XRR, $\rho_x(z)$ is proportional to electron density [3, 4]. In case of PNR, $\rho(z)$ consists of nuclear and magnetic SLDs such that $\rho^{\pm}(z) = \rho_n(z) \pm CM(z)$, where C = 2.853×10^{-9} Å^2 cm^3/emu, and $M(z)$ is the magnetization (in emu/cm^3) depth profile [3]. The sign +(-) is determined by the condition whether the neutron beam polarization is parallel (antiparallel) to the applied field and are represented by reflectivity, $R^{\pm}(Q)$. $\rho_n(z)$ and $M(z)$ can be inferred from $R^{\pm}(Q)$ often with less than nanometer resolution. Using PNR and XRR independently it is possible to extract exact chemical composition in binary alloys formed at the interfaces [4, 7]. Off-specular reflectivity (angle of incidence ≠ angle of reflection) provides detailed morphology of the surfaces and buried interfaces (in-plane structure) [3, 6].

We have developed a Polarized neutron reflectometer instrument (Fig. 1) at Dhruva reactor, Mumbai [8]. The instrument has been designed with a linear position sensitive detector, which helps to simultaneously measure the specular and off-specular neutron reflectivity over a large range of wave vector transfer parallel, Q_x [= $\frac{2\pi}{\lambda}\left(cos(\theta_i) - cos\ (\theta_f)\right)$, where λ is wavelength of neutron] and perpendicular, Q_z [= $\frac{2\pi}{\lambda}\left(sin(\theta_i) + sin\ (\theta_f)\right)$], to the sample's surface (Fig. 1 (b)). Here we present some of the important studies carried out by us using PNR.

(i) Investigation of Interface magnetization in Fe/Ge systems

Interfaces between ferromagnetic (FM) metals and semiconductors have attracted much interest because of their potential applications in electronic and ''spintronic'' devices. Growth of transition metal on semiconductor generally results in intermixing/interdiffusion at the interfaces, which decreases the magnetization of FM/semiconductor structures. We have investigated interface magnetization in Fe/Ge systems [9-12]. Here we present XRR and PNR study on a Si(substrate)/Ge/Fe/Ge trilayer system. We could quantify physical and magnetic roughness as well as morphology at the interfaces of the sample. Fig. 1(a) shows specular XRR from the sample. Which clearly suggest (Fig. 1(b)) a well-defined 3 layer structure of

the film with a roughness of ~ 9Å. Off-specular study on the same film suggested different level of inter-diffusion at the interfaces.

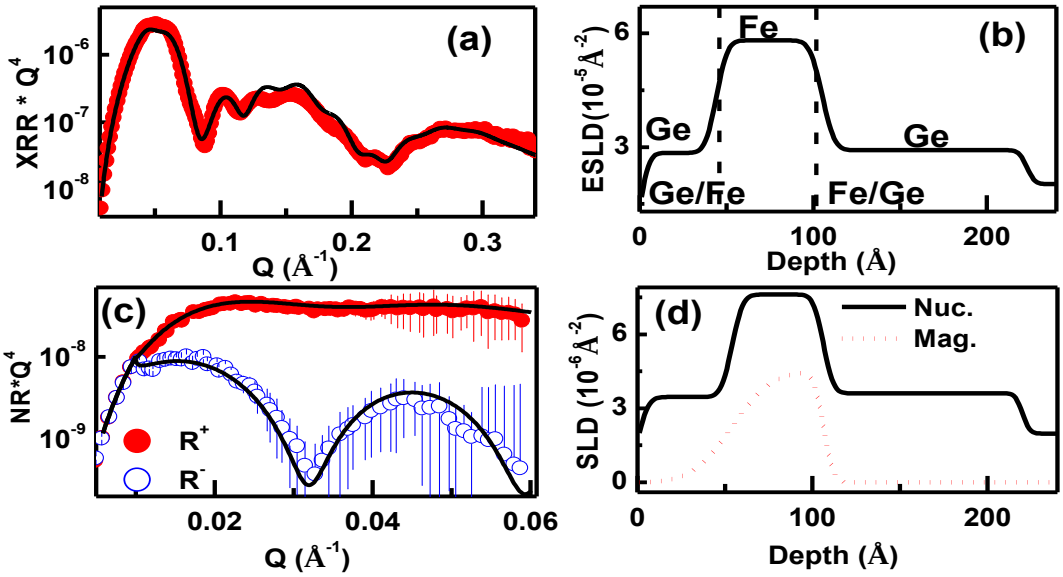

Fig. 2: (a) XRR data. (b) Electron scattering length density (ESLD) depth profile extracted from XRR. (c) PNR data. (d) Nuclear and magnetic SLD obtained from PNR data.

Specular PNR (Fig. 1(c)) measurement demonstrated the existence of different magnetic and physical density profile at interfaces in the sample [Fig. 2(d)]. The magnetic density profile was also asymmetric compared to the physical profile at the interfaces. This was attributed to different level of inter-diffusion at interfaces as suggested by off-specular XRR.

(ii) Kinetics of Intermetallic Alloy Formation at The Interfaces on Annealing

Ni/Ti multilayers have been studied extensively as ideal candidates for neutron optics components like highly reflecting mirrors, supermirrors, polarizers and monochromators, etc., because they have an excellent contrast factor for thermal and cold neutrons. Alloying of Ni/Ti at their interfaces in such composition-modulated samples show various interesting behavior, such as structural changes from crystalline to amorphous phase. In this study we have found that contrary to the previous observations, alloying at the interfaces of Ni/Ti multilayer samples may also progress through formation and growth of micro-crystals at the interfaces. We had prepared Ni/Ti multilayers on glass substrate by vacuum deposition [7]. The samples were annealed at 300 ^{0}C and 400 ^{0}C for a fixed time of 1.5 Hours.

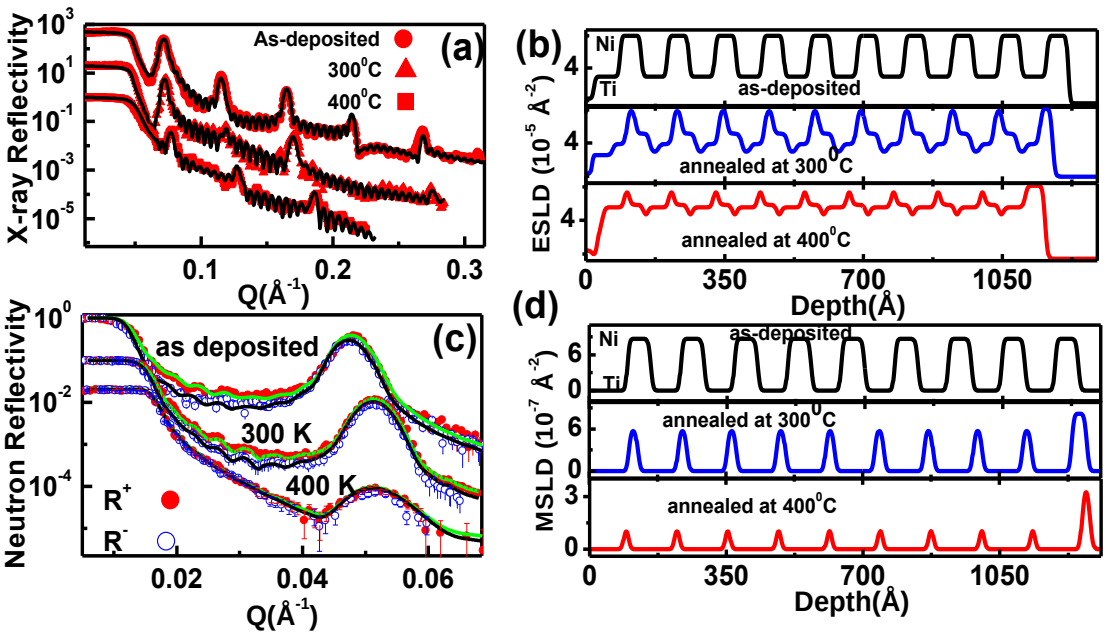

Fig. 3: (a) XRR data. (b) Electron scattering length density (ESLD) depth profile extracted from XRR. (c) PNR data. (d) Magnetic SLD obtained from PNR data.

Fig. 3(a) shows the XRR measurements from as-deposited and annealed Ni/Ti multilayers. Fig. 3 (b) is the extracted electron scattering length density (ESLD) depth profile suggesting modulation at interface on annealing. Fig. 3(c) and (d) show PNR measurements and corresponding magnetic SLD (MSLD) depth profile, respectively. We found that in these samples annealing produces crystalline phases at the interfaces and the grains have grown with annealing temperature, unlike the growth of amorphous phase discussed in previous studies. Growth of the micro-crystallites was confirmed from narrowing of x-ray powder diffraction (XRD) lines. The results suggested formation of Ni_3Ti alloy at interfaces on annealing which is crystalline and is also magnetically dead.

(iii) Interfacial mixing in as-deposited Si/Ni/Si thin film system

Extensive research over the past few decades has been devoted to the growth mechanism of metal silicides at the interface of metal/Si layered systems. On parallel lines numerous applications of silicides in the microelectronics industry have evolved based on metal/Si and silicide/Si interactions. This makes it important to study interdiffusivity and solid state reactions involving metal- semiconductor combinations, for a better understanding of interfacial modifications. Alloy formation due to interdiffusion across the interfaces of Si/Ni/Si system has been studied using the non-destructive and highly sensitive techniques of XRR and PNR [13].

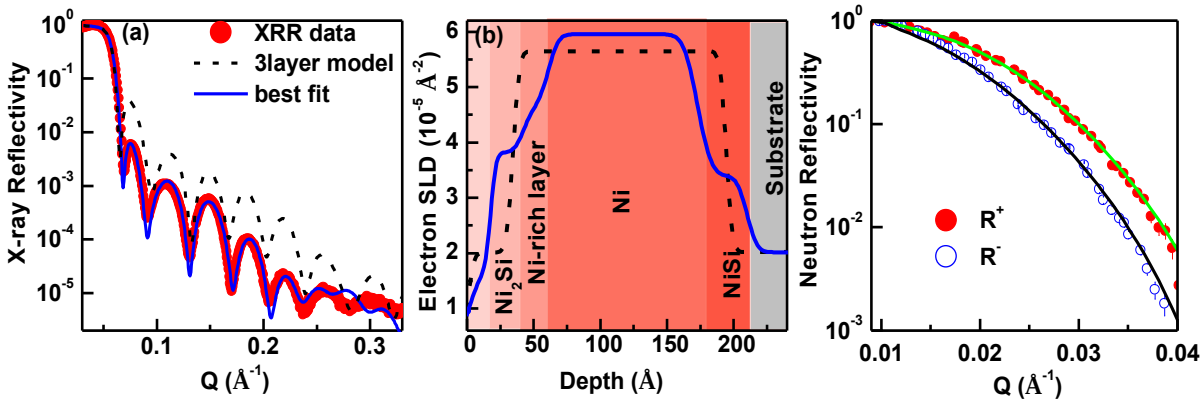

Fig. 4: (a) X-ray reflectivity from Si/Ni/Si system (b) electron scattering length density (SLD) depth profile extracted from XRR. (c) Polarized neutron reflectivity of the film.

Fig. 4(a) shows the x-ray reflectivity pattern from sample. Detail electron SLD profile extracted from XRR is shown in Fig. 4(b). Fig. 4(c) shows the PNR data from the sample. A quantitative assessment of the stoichiometry of interface alloys was obtained from the analysis of XRR and PNR data[4]. The alloy compositions on either side of the Ni layer could be closely associated with bulk Ni silicides. Additionally from PNR data fits, the magnetic moment per atom for all the compositions of the alloy layers estimated, were extracted. Combination of XRR and PNR techniques thus allowed us to detect both physical and magnetic structure of the layered sample with good spatial resolution.

(iv) Separation and Correlation of Structural and Magnetic Roughness in a Ni Thin Film by Polarized Off-specular Neutron Reflectometry

Magnetic roughness in addition to chemical roughness is supposed to play more important role to understand the interface magnetization multilayers. In this study the possibility of separating magnetic and chemical roughness in a Ni film through polarized diffuse neutron reflectivity (PDNR) was elaborated [14-15]. We measured PDNR data at Dhruva reflectometer from Ni film. The PDNR data were analyzed by us by after obtaining various PDNR amplitudes under Born approximation [14], which assumes the existence of a structural (chemical) and a separate magnetic boundary buried below it at each interface as shown in the inset (a) of Fig. 5.

Fig. 5 depicts the PDNR data for spin up (R^+) and spin down (R^-) neutron measured at fixed Q_z = 0.0252 Å^{-1}. Closed and open circles are the experimental data for spin up and spin down neutron respectively. We have collected the data centered on a specular peak beyond the total reflection region to remain in the weak scattering region where Born approximation is valid. For the off-specular data, we positioned the specular peak at one end of the detector, to reach largest possible value of the in-plane momentum transfer (Q_x), to test the quality of

fit over a large Q_x range. Over the entire Q_x range there is large difference in intensity between the up and down polarized beams.

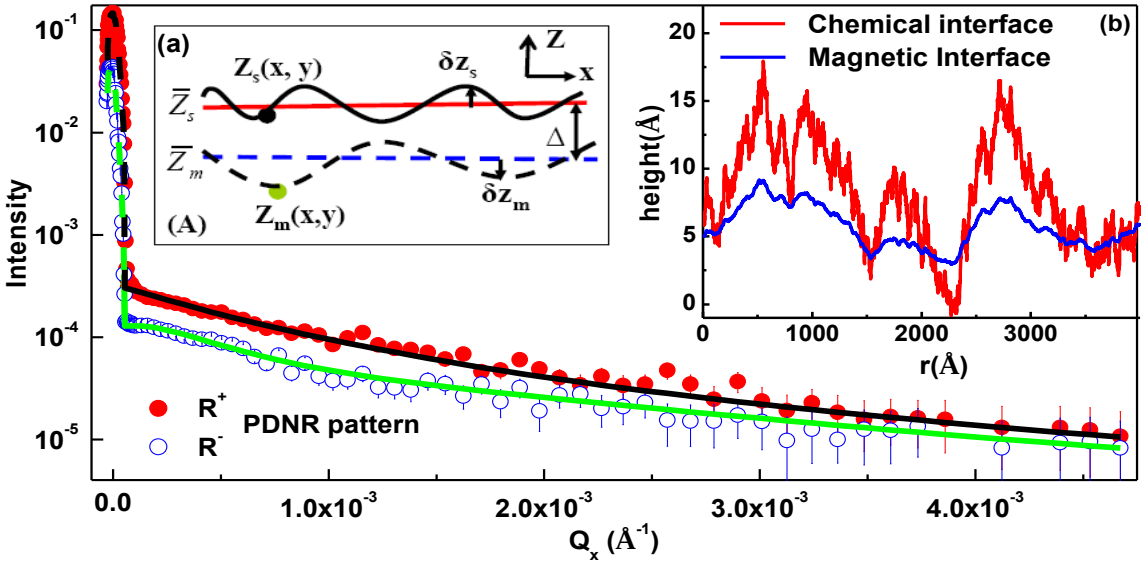

Fig. 5: Off-specular polarized neutron reflectivity measurements from the Ni film. Open circles and closed circles are the experimental data for spin up and spin down neutron at a Q_z = 0.0252 Å^{-1}. Continuous lines are the best fit. Inset (a) shows a cartoon of the structural (chemical) and magnetic boundaries at an interface. Inset (b) shows the height height fluctuation at chemical and magnetic boundary extracted from reflectivity data.

From the analysis of PDNR data, we have demonstrated that the magnetic boundary shows a higher in-plane correlation length with smooth surfaces. In this exploratory experiment and data treatment we have striven to demonstrate that PDNR can be used as a powerful tool to look at magnetic and chemical interfaces simultaneously.

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SANS study on nanostructure of thin-film-composite membrane for desalination and water purification

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Abstract

A typical 'reverse osmosis'(RO) or 'nanofiltration' (NF) polyamide membrane for application in desalination and wastewater treatment is formed by interfacial reaction between an aqueous solution of *m*-phenylenediamine (or piperazine) and a trimesoylchloride solution in *n*-hexane. In both the membranes, polysulfone films of about 40 μm thickness were used as supports over which thin polyamide layers of about 0.2 μm thickness was laminated. The small-angle neutron scattering profiles of the composite films suggested highly compacted nanometer-scale polyamides blocks which agreed well with the polymer nodular morphology observed by AFM. From dilute aqueous solutions of suspended polyamides a clear scattering from compacted nanometer-sized blocks of about 120-140 Å in radii was observed for both RO and NF polyamide.

Introduction

Fresh water is required for domestic uses, agricultural irrigation and industrial processes. Thin-film-composite (TFC) reverse osmosis (RO) and nanofiltration (NF) membranes are state-of-the-art membranes which are employed for production of potable (fresh) water by desalination and wastewater treatments [1, 2]. The TFC membranes are prepared by coating a selective polyamide layer over a porous polysulfone support and they provide an effective solution for producing potable fresh water from saline and wastewater in which the TFC RO polyamide membrane can reject (~99%) all the electrolytes present in saline water and the TFC NF polyamide membranes, which have slightly larger pores can selectively reject larger ions or molecules such as divalent electrolytes, dyes, proteins etc. The top polyamide membrane layer of the TFC is principally responsible for permeability and selectivity.

The understanding of the membrane materials are required to assess the membrane processes in order to develop membrane of improved properties. The membrane processes of the TFC membrane deal with species of sub-nanometer size ranges which means that the

scale of resolution of the observations should be in angstroms. Thus to observe real-time experiments such as transport process of a permeating species moving in the sub-nanometer pores of the TFC membrane has been very difficult even though there are few speculations to understand the transport phenomena based on model-based approaches. Small-angle neutron scattering (SANS) is powerful technique to understand the polymer chain nanostructure as well as the pore structure characteristics of the membrane in order to improve the membrane performance.

Experimental

The TFC RO polyamide membrane was prepared by initially immersing the polysulfone support film of about 40 μm thickness in a 2% (w/v) solution of *m*-phenylenediamine in water, followed by immersing into an *n*-hexane solution of 0.1% (w/v) trimesoyl chloride, which resulted in lamination of about 0.2 μm skin layer of polyamide over the surface of the polysulfone support [3]. For the preparation of the TFC NF membrane, piperazine instead of *m*-phenylenediamine was used [4]. For preparation of dilute solutions of polyamide, the above coating procedure was followed for coating the polyamide films over the surface of a glass plate. Immediately, the nascent polyamide films were washed off from the surface of the glass plate and collected in water. The immediately removed films were easily broken into flakes or pieces in water and thoroughly washed. These polymer flakes or smaller pieces were floated in water. The polymer sample in water was then vigorously shaken to break further the flakes into smaller pieces to finally form a colloidal-like suspension. Colloidal-like suspended polymer (7%, w/v) in aqueous solutions for the SANS measurements. The solution was quite homogeneous and falling of polymer particles to the bottom of measuring cuvette did not occur during the many hours of the scattering experiment. The SANS measurements from the membrane film samples or dilute solution preparations were taken on the SANS instrument at the Dhruva reactor, BARC, Mumbai, India. Throughout the data analysis, corrections were made for instrumental smearing.

Results and Discussion

The SANS patterns as shown in Figure 1 for both the RO and NF TFC polyamide-polysulfone composite membranes as well as the neat polysulfone support layer were measured to examine polyamide thin film structure over the polysulfone. The intensity of polyamide scattering, which was the difference of composite and neat polysulfone scattering intensities were treated using fits for a population of polydispersed spherical particles,

including structure factor arising from interactions between the particles only to generate initial approximations with reasoning, on how polyamide particles build skin layers of both the membranes. The broad correlation peaks of the difference SANS profiles over the *Q* range 0.0505 – 0.0555 $Å^{-1}$ corresponding to the length scale ($2\pi/Q$) of ~120 Å as shown in Figure 1 is a result of some structure having somewhat orderly arrangement of repeating units. There was a good agreement between the SANS data with the fits which suggests that these correlation peaks are due to the interparticle structure factor *S(Q)* generated by the interacting particles. Figure 2 shows a surface AFM image of the RO and NF polyamide membranes. The surface morphology of the sample as revealed by the AFM image shows a typical nodular morphology of polyamide membrane [5]. It clearly shows the inter-grown structures formed by the individual peaks. In case of the RO polyamide, the average surface roughness analysis from 20 µm x 20 µm image frame was found to be about 140 nm. Further in case of RO polyamide, it was observed that each bigger peak was comprised of numerous smaller peaks in which individual peak surface roughness was apparently as about 10-20 nm only. Inter-grown nodular structures formed by about 40-50 nm were also observed in case of NF polyamide membrane. Thus, AFM images revealed highly compacted nodular structures of the TFC polyamide membranes.

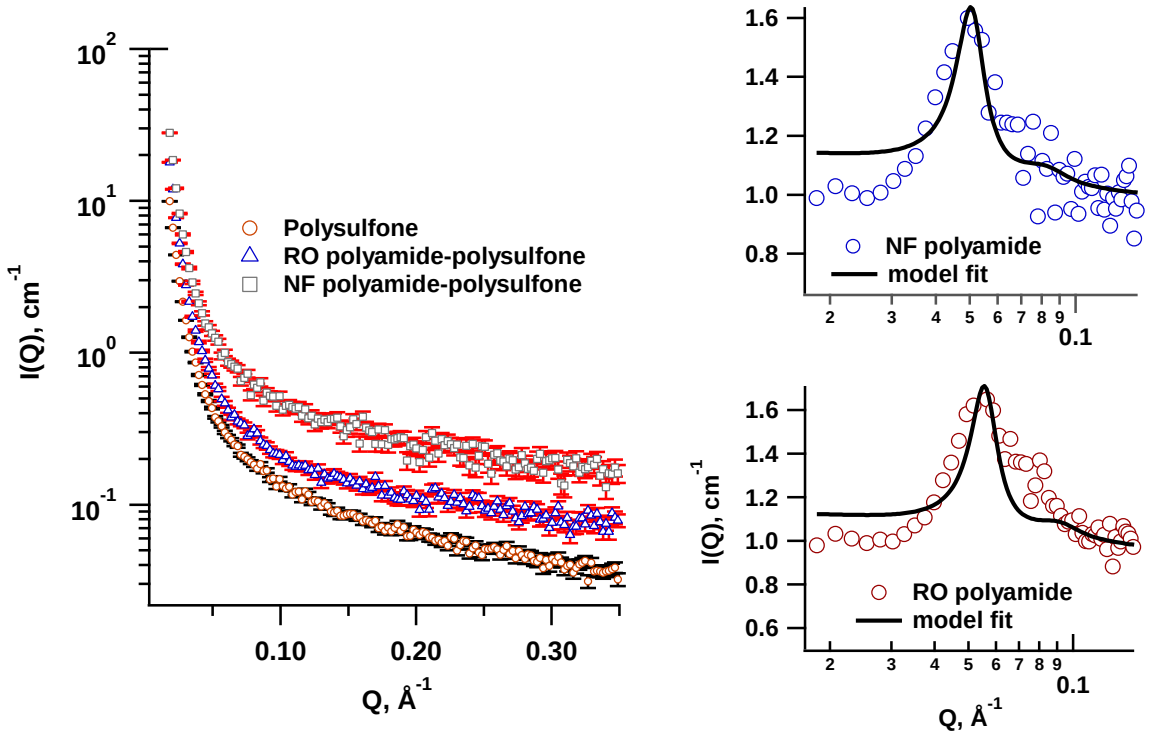

Figure 1. SANS profiles of composite membrane films of RO polyamide-polysulfone and NF polyamide-polysulfone along with the profile of polysulfone support film (Left). The calculated intensity of neutron scattering as a function of scattering vector from the top RO and NF polyamide layer of the composite films (Right)

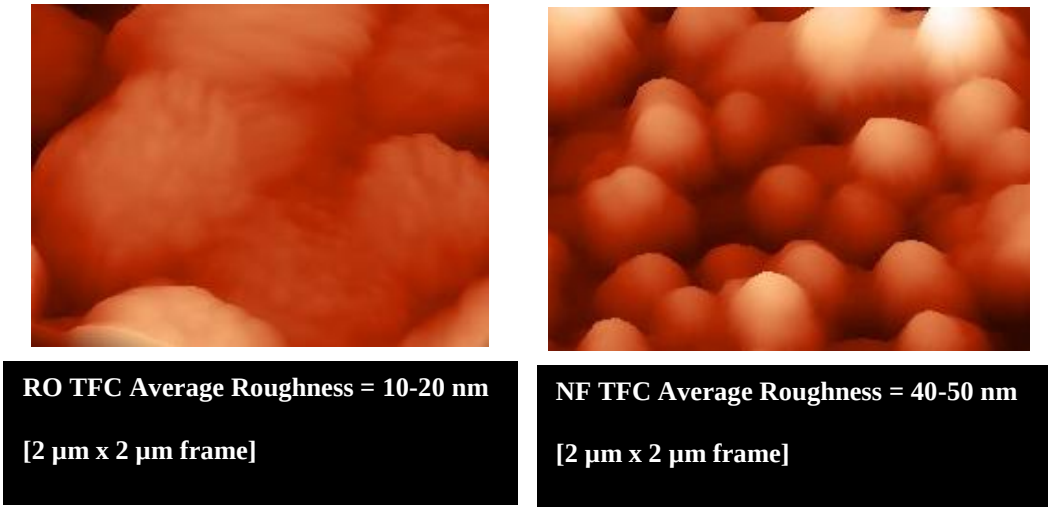

Figure 2. Top surface AFM images of the RO and NF TFC membranes

In order to examine the scattering contributions from the primary polyamide material that make a compacted membrane layer of interest, SANS measurements were also performed on the colloidal-like suspension dilute solution systems which were prepared by dispersing the nascent polyamide film washed off from a glass substrate in which the preparation of the polyamide on the glass substrate was the same as that of the membrane coating process. Figure 3 shows SANS profiles from the RO and NF polyamide solution samples which were fitted well with the sphere form factor fits well although there was no pronounced minimum due to the polydispersity. The average size of scattering particles given by the sphere form factor fits are in the range of 128-138 Å with the median radius ranging from 106-119 Å and the polydispersity fraction, σ of 0.46. The details of analysis can be found in our earlier publication [6].

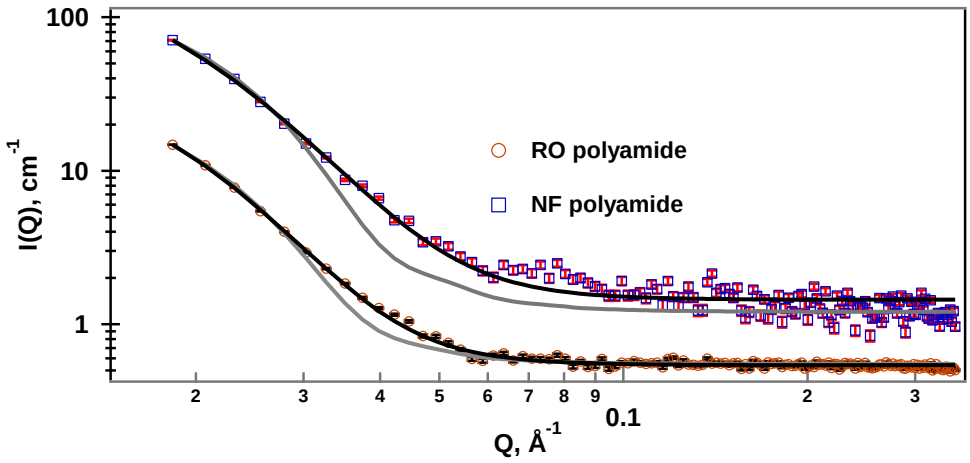

Figure 3. SANS profiles along with fits of the RO (bottom profile) and NF (top profile) polyamide samples suspended in water. Grey line: spherical form factor fit; line drawing: polydispersed spherical form factor fit.

Conclusion

A typical RO or NF polyamide structure formed by interfacial reaction between an aqueous solution of *m*-phenylenediamine or piperazine and *n*-hexane solution of trimesoyl chloride possesses were comprised of nanoscale building blocks as revealed by Small angle neutron scattering study on the RO and NF polyamide membranes. The nanoscale building block of the RO polyamide membrane in average has similar size with that of the NF polyamide membrane.

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Conferences and Workshops 2013

July 2013

- NOP&D-2013: International Workshop on Neutron Optics and Detectors (ICNS satellite meeting)
  July 2-5, 2013, Munich, Germany
- ICNS 2013: International Conference on Neutron Scattering
  July 7-11, 2013, Edinburgh, UK

August 2013

- 15th Annual National School on Neutron and X-ray Scattering
  August 10-24, 2013, Argonne and Oak Ridge, USA
- 12th PSI Summer School on Condensed Matter Physics
  August 17-25, 2013, Zuoz, Switzerland

September 2013

- 17th Laboratory Course Neutron Scattering
  September 2-13, 2013, Jülich and Garching, Germany
- 13th Oxford School of Neutron Scattering
  September 2-13, 2013, Clarendon Laboratory, Oxford, UK
- 11th International Conference on Biological Synchrotron Radiation
  September 8-11, 2013, Hamburg, Germany
- 3rd Joint User Meeting at PSI: JUM@P 2013
  September 18-20, 2013, PSI Villigen, Switzerland

October 2013

- JCNS workshop 2013: Trends and Perspectives in Neutron Scattering: Magnetism and Correlated Electron Systems
  October 7-10, 2013, Tutzing, Germany
- COM 2013 incl session on Applied Neutron Scattering in Engineering and Materials Science Research
  October 27-31, 2013, Montreal, Canada

December 2013

- Neutron Scattering and X-Ray Studies for the Advancement of Materials at Thermec 2013
  December 2-6, 2013, Las Vegas, USA

February 2014

- Conference cum School on Neutron Scattering
  February 4 – 8, 2014 (Pre-Conference School), BARC, Mumbai
  February 10-12, 2014 (Conference), IISER, Pune